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THE PRECIPITATION PROCESSES OF Cu-Zr-Cr ALLOYS

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16. Abstract

precipitation processes of a Cu-Zr-Cr alloy. The hardness measurements, the measurements of specific heat and electrical resistance were carried out to examine the effects of Zr and Cr on the Cu-Cr binary alloy. The specimens were solution treated at 950°C and quenched into ice water. Subsequently they were cold rolled at a 80% reduction and aged under various conditions. In the study, the precipitation processes during aging at temperatures elevated at a constant rate of 2°C per minute were examined. [illegible] were [illegible]. In the case of the binary Cu-Cr alloy, the precipitation [illegible] at 440°C and [illegible] precipitation started at about 525°C. The [illegible] was about 0.4 at% under the limit of Cu-Cr alloy. In the ternary specimens, the [illegible] precipitation [illegible] and Cr appeared at about 350°C and 425°C, respectively. The [illegible] with that of the binary alloy, with the exception of the above mentioned [illegible]. It was found that the above mentioned [illegible] of the [illegible] of the [illegible] and [illegible] of the binary alloy [illegible] to the [illegible] and [illegible] [illegible] [illegible]

THE PRECIPITATION PROCESSES OF Cu-Zr-Cr ALLOYS*

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Hisashi Suzuki and Motohiro Kanno**

Hisashi Suzuki and Motohiro Kanno : Precipitation Processes of Cu Zr-Cr Alloys.
Precipitation processes of a Cu Zr-Cr ternary alloy have been investigated by means of specific heat and electric resistance measurements, comparing with those of Cu-Zr and Cu-Cr binary alloys. Specimens were solution-treated at 950 C and quenched into iced water. Subsequently they were aged or cold-rolled about 83% and then aged under various conditions. In this study, the precipitation processes during aging at temperatures elevated at a constant rate of 2 C/min were examined in detail. The results obtained were as follows: 1) In the as-quenched specimen, Cr precipitate appeared first at about 440 C and then Cu_3Zr precipitate occurred separately at about 520 C. The former temperature was about 15 C higher than that of Cu-Cr alloy. In the worked specimen, the order of precipitation was reversed, that is, Cu_3Zr and Cr appeared at about 370 C and 425 C, respectively. 2) The two sets of precipitation temperature observed in each case of the ternary alloy agreed well with that of each binary alloy, with the exception of the above-mentioned temperature of Cr precipitation. 3) It was shown that the above results could satisfactorily be explained by the precipitation diagram for T-T-T diagram of the ternary alloy which was established qualitatively by superposing of four curves, two curves corresponding to homogeneous and heterogeneous precipitations of Cr and the other two curves of Cu_3Zr. The property change during isothermal aging can also be well explained by using the diagram.

I. Preface

Concerning age hardened type Cu-Zr-Cr alloys, the relationships between the characteristics and their makeup or heat treatment [1]-[3], the even numbered sectional situation diagram for copper [4], their strength, the relationship between heat resistance and aging structure [5], and the reversion phenomenon have been investigated and all of the characteristics of the resulting main alloys and the aging phenomenon have become clear. It is still not known, however, for the two types of precipitants (Cu_3Zr,Cr) that result if there is a precipitation sequence or not, the nature of the speed of precipitation and if they change through the processing. Also, it seems that normally for isothermal aging, the conductance does not recover well, but it is not known whether this is a hindrance or not. It is thought that if this was known, the alloy's characteristics would be even

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*** Nos. in margin indicate pagination of foreign text.

better known and a new field of application would open up. Therefore, in this report the precipitation processes (precipitation sequence, speed, etc.) have been pursued by means of specific heat and electrical resistance measurements.

II. Materials and test methods

Since for the testing of ternary alloys, it is necessary to investigate the two alloys of zirconium copper and chromium copper (below abbreviated Cr copper, Zr copper), besides the Cu-0.54 % Zr-0.50 % Cr alloy, we used as a sample 0.30 % Zr copper and 1.0 % Cr copper. The materials were prepared in the same manner as those in the previous reports [4]-[6] with a plate of a 0.5 mm thickness. In order to see the effects of cold treatment, after solution treating a 3 mm thick plate, we cold-rolled it to about 83%, making it the same 0.5 mm thickness. For all of the samples, solution-treatment was set at 950°C.

For the electrical resistance measurements, we used a sample 0.5x10x170 mm, the times values of the quenching and aging were set at the various aging temperatures and measured continously. Measurements were carried out at temperature up conditions the same as the specific heat measurements noted below.

Normally, when doing specific heat measurements block samples are used but in order to investigate the effect of processing on the precipitation processes (this being one of our main purposes), we used plates all with a thickness 0.5 mm layered onto form materials 20 mm ø x 25 mm as the test sample, and carried out measurements at low pressure AR atmosphere (insulation method, temp. rise speed 2°C/min). For determining the applicability of the layered sample materials, we used a block sample cut off of a regularly marketed 1.0% Cr copper bar.

III. Results

First, we looked into the change in resistance at the time of aging at 400°C and 450°C that was carried out on all samples immediately after solution treatment and quenching. The results are in Figure 1. It is natural that precipitation speed would be as fast as the high temperature for the various alloys at 400°C and 450°C. But we learned by looking at these same temperatures that there are differences between the alloys precipitation speeds.

Here we do not see a differential curve but in the initial /364
stage of precipitation, the speed for Cr copper is fastest. For example, at 450°C, it completes within one hour its most intense transformation. At the same time, the speed of precipitation for Zr copper and Zr-Cr copper is slow and it is certain that the precipitation continues for a long time.

In Figure 2 is an example of the results of 400°C aging for processed materials. Compared to the 400°C unprocessed materials in Figure 1, the speed of precipitation for Cr copper hardly changes. For all but Zr copper and Zr-Cr copper the speed in the initial stages is strikingly faster. In other words, the speed in the initial period for these two alloys is faster than that of Cr copper. For Figure 2, if we go on to carry out long term aging, the Zr copper finishes its transformation after three hours but on the other hand, the Cr copper and Zr-Cr copper's transformation, almost like the processed case, continues for almost 50 hours. Of course, for 450°C again, the resistance transformation completion time is compressed, but the tendency for 450°C aging 15 for it is to be nearly the same. From the above it becomes clear that (1) the precipitation in the initial stages for Zr copper and ternary alloys is facilitated by processing; (2) for Cr copper this hardly changes at all; (3) in other words, in the case of unprocessed materials, the aging for Zr copper and ternary alloys is slower and longer than that for Cr copper but

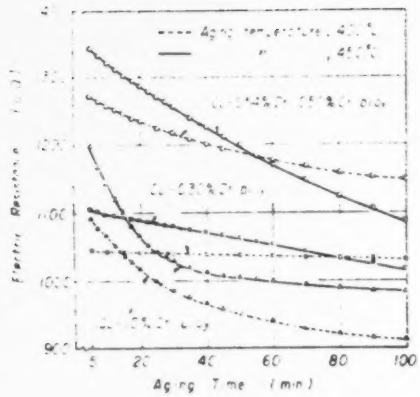

Fig. 1 Changes of electric resistance of Cu-Cr, Cu-Zr and Cu-Zr-Cr alloys as a function of aging time. Specimens were solution-treated at 950°C, quenched and subsequently aged at 450°C or at 400°C. Electric resistivity measurement was continuously made at the aging temperatures.

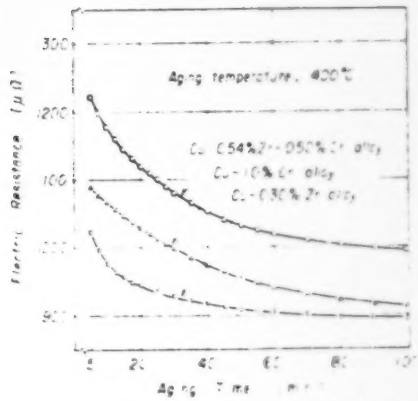

Fig. 2 Changes of electric resistance of the alloys cold-rolled 85% as a function of aging time. Specimens, solution-treated, quenched, cold-rolled and aged at 400°C.

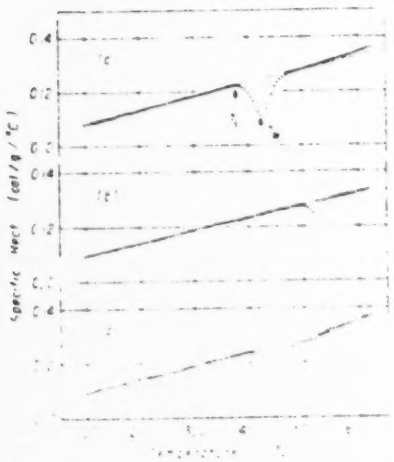

Fig. 3 Specific heat vs. temperature curves of Cu-Cr, Cu-Zr and Cu-Zr-Cr alloys. Specimens solution-treated at 950°C, quenched and subsequently heated at the rate of 2°C/min.
a: Cu-2.0% Cr alloy, b: Cu-0.3% Zr alloy, c: Cu-0.54% Zr-0.05% Cr alloy.

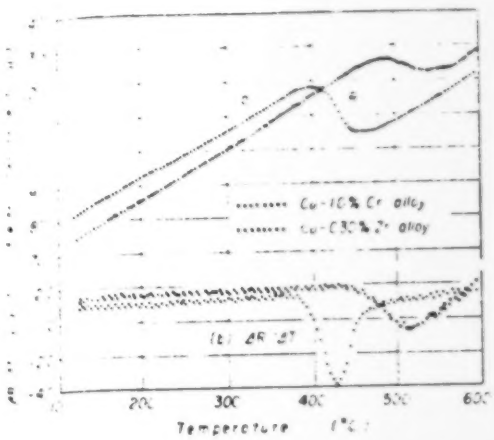

Fig. 4 Electric resistance vs. temperature curves, a, and the differential curves, $\Delta R/\Delta T - T$ curves, b, of Cu-Cr and Cu-Zr alloys. Specimens, solution-treated, quenched and subsequently heated at the same rate as in Fig. 3.

if processed Zr copper becomes the very fastest, even (illegible) faster than Cr copper or ternary alloys. It is ascertained that at the aging temperature noted above the aging of terniary alloys depends not on whether or not it has processing but on the (illegible).

Still unknown is through what kind of precipitation process the two types of precipitation brought about the resistance transformation noted above. With this in mind, we carried out the following Cp measurements. First, we compared the results with layered samples and those of lump samples. According to this, the results for the layered samples were somewhat smaller than the Cp change in orders and the growth in Cp brought on by the temperature rise became somewhat large (it is thought that the cause is the advantages and disadvantages of the layered sample material), but for both layered and lump samples the temperature when the change was rapid was the same. So even for materials beyond Cr copper, it is thought that layered sample materials do not create a hindrance in the pursuit of the Cp change and we decided from here on to employ layered samples in all tests. Figure 3 shows the results of the case when solution treated samples were immediately subjected to temperature up. According to this, the temperature (T_s) where the beginning standard line first is pulled in a lower direction for (A) Cr copper is about 380°C, and the temperature where the quantity of heat generation (precipitation quantity) is highest is 425°C (T_m). After the heat generation had continued to the area of 490°C, there was a mild heat absorption over approximately 500°C. For Zr copper (b), at a temperature higher than Cr copper 500°C-530°C, we see heat generation but the quantity is quite small (the solid-liquid quantities of Zr are at 950°C about 0.1% and are smaller than Cr's 0.26%) [4]. For (3) ternary alloys $T_s \simeq 400°C$, $T_n \simeq 440°C$ and even at 500°-530°C, a small heat generation is produced. Corresponding the two heat generations in the ternary alloys with the results of the binary alloys will give the various precipitations of Cr, Cu_3Zr.

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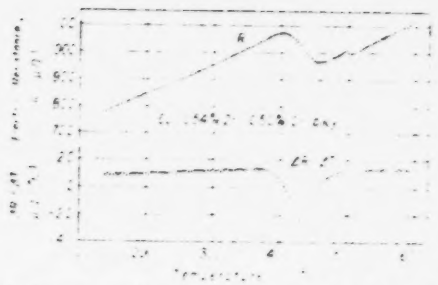

Fig. 7 Electric resistance and $\Delta R/\Delta T$ vs. temperature curves in Cu-Zr-Cr alloy. Heat treatment and testing conditions were the same as in Fig. 4

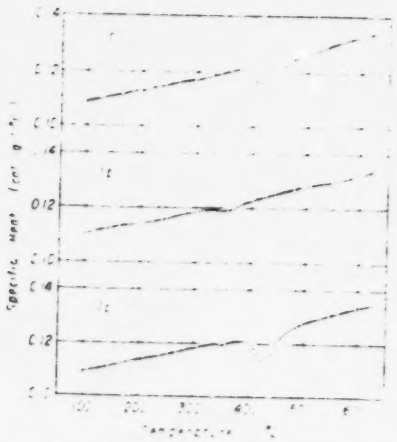

Fig. 9 Specific heat vs. temperature curves of various Cu-Cr alloys cold-rolled about 83%, showing similar to the curves obtained in as quenched specimens in Fig. 3

a Cu-1.0% Cr alloy, b Cu-0.30% Zr alloy, c Cu-0.54% Zr-0.50% Cr alloy

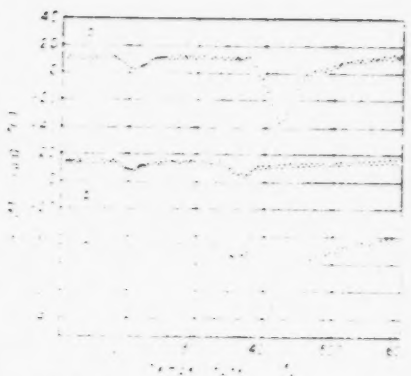

Fig. 7 $\Delta R/\Delta T$ vs. temperature curves corresponding to Fig. 6

a Cu-1.0% Cr alloy, b Cu-0.30% Zr alloy, c Cu-0.54% Zr-0.50% Cr alloy

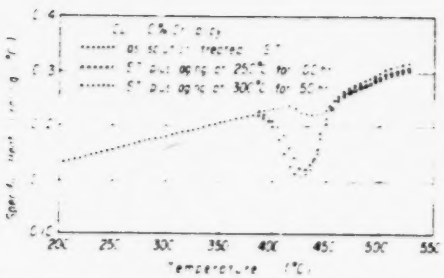

Fig. 8 Variations in specific heat vs. temperature curves of Cu-Cr alloy as a function of specimen conditions before heating specimens

Next,the specific heat measurement was corresponded and the electrical resistance was measured under the same temperature up conditions. Figure 4 shows the results for Cr copper and Zr copper; Figure 5 that of the ternary alloys. For both cases,the curve for the change ratio $\Delta R/\Delta T$ and resistance R corresponded to the change in the specific heat curve in Figure 3. This is natural because of the pluses and minuses /365
of the changes in electrical resistance brought on by the precipitation or the rehardening-liquifying. Incidentally, if we pay attention to the $\Delta R/\Delta T$ curve, the two dips that look like the curve of the ternary alloy stack up, matching the two $\Delta R/\Delta T$ curves in Figure 4 and they go along with the dips seen in Figure 3(c). It becomes clear that the dips of the high or low temperature are based on the various precipitations of Cr or Cu_3Zr. According to this, if we do a solution-treatment, quenching and temperature up aging, with ternary alloys the precipitation of Cr takes precedence and that of Cu_3Zr is late. The two types of precipitants precipitate separately. Also the precipitation temperature of Cr is about 15°C higher than that of Cr copper.

Since the result of unprocessed samples are known, we proceed to test under the same conditions with processed materials. Figure 6 has the results for the specific heat measurements. In a) Cr copper, a small temperature generation started at the first 180-200°C and after that there was a large heat generation much like that shown in Figure 3 at 425°C. For (b) Zr copper,there was a small heat generation on the area of 200°C and on the area of about 370°C it became somewhat large. The temperature of the heat generation when compared to the Figure 3(b) temperature of 520°C is strikingly low. For the ternary alloys (c), just the same as at 200°C, there is a very small heat generation, and in correspondence with (b), at about 370°C there is a small heat generation. Finally, in correspondence to (A) at 425°C, there was a large heat generation. In other words, this 425°C temperature that shows a large heat generation gives a precipitation

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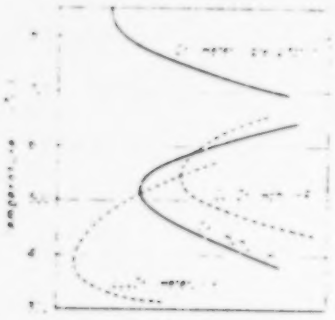

Fig. 6 Schematic T-T-T curves of Cu-Zr-Cr alloys. [illegible] of C curves were drawn [illegible] and heterogeneous [illegible] the kinds of precipitates.

Fig. 7 Transmission electron micrograph of Cu-0.54 wt% Zr-0.6% Cr alloy. Specimen was solution-treated at 950°C for 1 hr, quenched, cold-rolled about 20%, and subsequently aged at 475°C for 1hr, at 500°C for 1hr.

temperature for Cr in the ternary alloys but the temperature in this case is almost the same as the precipitation temperature of Cr occurring in Cr copper.

Figure 7 has the results that demanded the $\Delta R/\Delta T$ for the three types of sample materials. As mentioned before, this $\Delta R/\Delta T$ curve corresponds well to the Cp curve in Figure 6. Since the change seen in the area of 180-200°C is not found in unprocessed materials, it is thought th t it is basic to the recovery of the processing organization. If we go ahead and compare Figures 3 and 5 with Figures 6 and 7, the precipitation of Cu_3Zr that is in Zr copper or the ternary alloys improves by way of processing and it becomes clear that the precipitation /366 temperature descends even lower than that of Cr. That the precipitation temperature of the Cr in the ternary alloys is the same as Cr copper is also well expressed by the $\Delta R/\Delta T$ curve.

Next, we consider if there is mimic stability appearing in the aging process or not. It has been reported that these three types of alloys show mimic stability but even after looking at these reports, the extreme Cp or resistance that would be thought of as the basis for these results is not to be found. Since the reversion phenomenon is present in the three alloys [6]

this point was investigated a little more closely. We carried out Cp measurements on samples completely pre-aged (treatment to increase the reversion quantity [6]) at low temperatures. For Cp copper, the example is seen in Figure 8. Due to the pre-aging T_s, T_m pulled somewhat to the side of high temperature and the quantity heat generation decreased. For such aged alloys as Al the heat absorption that is the basis of the reversion of the mimic stability (which appears in the early stages of the heat generation reaction [8], [9] was not seen at all.

The heat absorption, not being found before the heat generation, was the same for Zr copper and the ternary alloys. Also carried out were the resistance tests, but the change that is thought to be the basis for this could not be detected in any case. The lack of heat absorption before the heat generation was the same as the Zr copper and the ternary alloys. According to these results, there is no corollation of mimic stability in ternary alloys, Zr copper or Cr copper. Also, it is understood that only when doing a normal, continuous temperature up is there no appearance of the reversion phenomenon. Because the previous report [10], which produced the reversion phenomenon by increasing the temperature of the Cr copper in stages and through annealing is limited to one type of case involving discontinuous heating, the results that come from thinking in the following manner are natural.

IV. Observations

If we summarize the main points of the results obtained above we get the following. Separation of the precipitation processes cannot be achieved with isothermal temperature aging. Only after temperature up aging can we first begin to have separation, but the two types of precipitants precipitate separately and if processed, the precipitation of Cu_3Zr improves remarkably, becoming easier for precipitation than Cr. This tendency is the opposite of the situation involving unprocessed materials. It is thought that this kind of precipitation process will appear even with

isothermal aging.

On the other hand, the precipitation temperature of Cr is, like Cu_3Zr, not transferred by processing and is thought to be fixed, but when there is no processing, it precipitated at a temperature even a little higher than Cr copper. For other continuous heatings besides this, no reversion phenomenon appeared and therefore it is thought that these alloys do not produce a mimic stability. This is one of the main results of this experiment.

It is important that among these results the relationship between the precipitation processes and processing is not the only point having meaning. We can also understand the characteristics of the alloys. Therefore, below we have carried out observations about these main points. According to the Cr copper T-T-T curve test results, the heterogeneous reflection arch was approximately 500°C and the heterogeneous reflection arch was about 850°C. This shows that as opposed to the homogeneous precipitation of the low temperature part of Cr copper, there is little effect from the rise in the dislocation density due to processing. At the same time,the same curve for Zr copper has not yet been observed but when it is isothermically aged with unprocessed materials, it is above 500°C [2] and with processed materials it is above 350°C and recovers its conductivity suddenly [3]. Cu_3Zr homogeneously precipitates [5] quite strikingly and considering the results of the temperature rise aging mentioned above, it is thought that the heterogeneous precipitation deflection arch of Zr copper is between approximately 350-400°C and the homogeneous precipitation deflection arch is between 500-550°C. Also, according to the results of this experiment, in the precipitation of ternary alloys the Cr and Cu_3Zr precipitate separately and due to this the reciprocal action of the Cr elements and the Zr elements will not be stronger than this and the precipitation of the ternary alloys could be thought of as stacking up and matching of those binary alloys.

(On top of that, as example alloys formed of the two types of precipitation of the ternary alloy, we have Al-Cu-Ag [13] and Al-Cu-Mg [14]).

It is thought from the observation above that the T-T-T* curve is like that shown in Figure 9 (considering the two types of precipitates separately). However, in this diagram the points where the precipitation temperature of the unprocessed material of Cr is not the same as that of Cr copper is ignored. With this we observed the relationship between this diagram and the results of our experiments. When heating the unprocessed materials of the ternary alloys, at a low temperature of 350-400°C first the Cu_3Zr precipitated heterogeneously,but because the dislocation density is low the precipitant quantity is low. Therefore, it cannot be detected. When the temperature is above 400°C the homogeneous precipitation deflection arch temperature comes close and the heterogeneous precipitation can be detected, if we continue to raise the temperature. Finally, the Cu_3Zr will be able to homogeneously precipitate and it is thought that these two types of precipitants had an effect changing Cp and R. For processed materials, because there was an increase in the quantity of precipitates from the homogeneous precipitation of Cu_3Zr, first the heterogeneous precipitation of Cu_3Zr and then the homogeneous precipitation of Cr was detected. Finally, it was thought that the precipitation temperature of Cu_3Zr moved to a lower temperature than that of Cr.

The results of the isothermal aging of unprocessed materials shown in Figure 1 can be understood in the following way: The Cu_3Zr that appears first (though heterogeneous precipitation)

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*The T-T-T in this case is not the so-called transformation start or completion showing type, it is a line diagram which has the precipitation set (the quantity of character transformation due to that is set); therefore it is a line diagram showing the criterion for judging the speed of precipitation.

is very small. Therefore, the homogeneous precipitation of Cr for the initial period of aging takes priority and it is difficult for the homogeneous precipitation of Cu_3Zr to occur. Thus, the precipitation speed in the initial stages of precipitation for Cr copper and tenary alloys is quite a bit faster than that for Zr copper. But in the later stages, the Zr copper and ternary alloys which are related to the Zr are slow and cause the resistance reaction to extend to a long period. It is not necessary to explain that for the aging of processed materials shown in Figure 2 the speed in the initial period for Zr copper and ternary alloys is faster than that of Cr copper and that it is slower than the speed in the latter period of Cr copper and ternary alloys.

In photo 1 is shown the structure of ternary alloy processed materials (in order to better observe the structure, the processing rate was put at 20%). According to this, at 400°C x 1 hr. the heterogeneous precipitation of Cu_3Zr is by far the most excellent and Cr cannot yet be seen, but at 500°C x 1 hr. the heterogeneous precipitation of Cu_3Zr and the homogeneous precipitation of Cr both can be seen, clearly supporting Figure 9.

Put another way, if we establish the ternary alloy T-T-T curve to be like that in Figure 9, the aging process of ternary alloys will change appropriately to the presence or absence of processing and the level of the aging temperature, and we can clearly understand that these conditions play a direct role in influencing the resistance transformation in the isothermic aging. Next, concerning the fact that the precipitation temperature of Cr in unprocessed ternary alloys rises more than in Cr copper, when unprocessed ternary alloys are aged normally. This is of particular interest because it is thought that the distribution of Cr precipitates is finer than that of Cr copper and the strength level corresponds with the temperature rise [4]. The cause could be both the Zr seizing the excess vacancy or the direct reciprocal action of the Zr and Cr elements. But the

precipitation of Cu_3Zr in processed materials comes first and as a result the precipitation temperature of Cr becomes the same as Cr copper. Another result of this is that if the Zr element has a vacancy capturing action, the vacancy is collected in the Cu_3Zr that precipitates in the beginning, so that even if the precipitation of Cr which is formed after that becomes delayed it seems to be all right. However, because this is, in reality, not the case the Zr element brings on a reciprocal action on the Cr element for the Cr precipitation occurring in the ternary alloys (unprocessed materials), and it is thought that perhaps it is checking the atomic formation and development of Cr. For Figure 9, if we keep this point in mind, the homogeneous precipitation curve of Cr in the middle of the diagram moves according to the presence or absence of processing approximately 15°C. This change has no effect on the above theory.

Lastly, we would like to turn to the reversion phenomenon of these alloys. First, we can learn that the lack of reversion in the continuous temperature up action as a result of there being no information of a mock stability was due to the effect of such G.P. (expansion unknown) molded alloys as the Al alloy. The problem is that there is reversion at a fast temperature up and there is none at continuous temperature up. The former has been used for a long time at a critical diameter explanation [6], [15], and can be explained recently by the Pashley model [16] and others related to the second stage of aging. However, the latter does not necessarily seem to be so well explained. If we try to apply the Pashley model to the latter we get the following: Because this model postulates kinetically the particles formed in the first stage of aging stabilize or negate thermodynamically. The particles formed in the first stage of aging, as the condition for reversion no matter how small they are, necessitate the appointed temperature difference between the first and second stages of the aging temperature. Therefore, in an experiment like this one where the temperature up is continuous, the conditions above are not fulfilled and it can be explained. However,

strictly speaking, this is present also in the speed of temperature up. Therefore it is necessary to test this in more detail in the future.

V. CONCLUSIONS

Paying special attention to the two types of precipitants (Cr, Cu_3Zr) that are formed in Cu-Zr-Cr alloys and testing the precipitation processes (mainly inside the temperature up aging) using electrical resistance and specific heat measurements, we gained the results listed below.

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(1) By carrying out temperature up aging at 2°C/min with unprocessed materials, the precipitation of Cr, first at about 440°C, and of Cu_3Zr at the higher temperature of approximately 520°C, occur separately. The precipitation temperature of Cr was about 15°C higher than that of Cr copper. The Cu_3Zr precipitation with processed materials progressed markedly; the precipitation temperature was about 370°C and the temperature for Cr was approximately 425°C (the same as that of Cr copper).
(2) The precipitation temperature of both types of precipitants in ternary alloys almost perfectly stacks up and matches the precipitation temperatures of the ternary alloys whether it has been processed or not. For unprocessed materials, the precipitation temperature of Cr is a little higher than that of Cr copper and this is due to the reciprocal action of the Cr elements and the Zr elements. (3) By hypothesizing using the T-T-T curve of ternary alloys, we can clearly explain the precipitation process of the two types of precipitants in ternary alloys. Of course, this curve is made from the two curves relating to Cr and Cu_3Zr and for each curve there is a heterogeneous and a homogeneous precipitation deflection arch. (4) We also learn that generally if this curve is used, the period of resistance transformation that occurs during the isothermic aging of ternary alloys is extended. Besides this, matters concerning the absence of a reversion when there is continuous heating and the fact that

there is no formation of mock stability in these alloys are also mentioned.

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